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Topical Report,

FRACTURE TOUGHNESS OF CVD ZnS

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ABSTRACT

The fracture toughness of CVD ZnS was measured by a modified expanded ring test technique to be

$$0.75 \pm 0.01 \text{ MPa m}^{1/2}$$

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Introduction

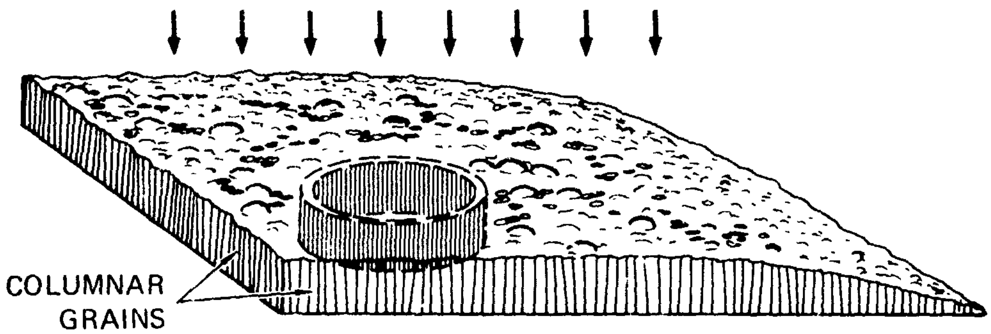
As part of a research program to define the material properties governing the impact erosion resistance of ir windows and radomes, we have measured the plane strain fracture toughness K_{Ic} of chemical vapor deposited zinc sulfide by a precise method, a modified expanded ring technique. This topical report describes the procedures and results.

Specimens

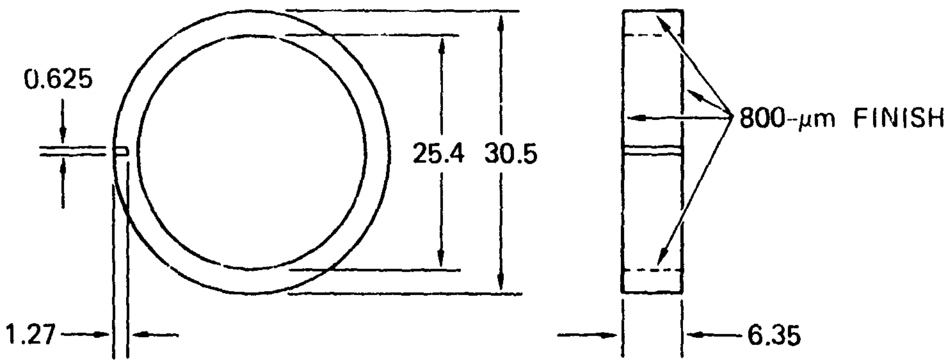
Chemical vapor deposited zinc sulfide (CVD ZnS) produced by Raytheon Corporation under contract to the Air Force Materials Laboratory (AFML) was supplied in as-deposited plate form through the kind cooperation of Mr. Lawrence Kopell (AFML) and Dr. James Pappis (Raytheon). The microstructure consisted of columnar grains oriented roughly parallel to the direction of vapor deposition (normal to the plane of the plate, Figure 1). The grains had average cross sectional diameters of 7 μm and aspect ratios of about 8. Six specimen rings, 25.4 mm inside diameter by 30.5 mm outside diameter by 6.35 mm high, were machined from the plate with the height dimension parallel to the growth direction (see Figure 1). The estimated finish on all surfaces was 800 μm. A flat-bottomed notch was introduced from the outside surface along a diameter to a nominal depth of 1.27 mm using a 0.25-mm-thick diamond wheel at 1000 surface meters per minute. To ensure accurate notch orientation and to reduce notch damage, we designed a special holding jig, and accomplished the notching in a number of 0.05 mm passes. Root radii were estimated to be 0.15 mm.

Experimental Technique

The expanded ring test technique for determining fracture toughness is basically the same as the technique used to determine precise tensile properties for brittle materials.[1] Ring-shaped specimens are loaded by increasing the hydrostatic pressure in a rubber annulus in contact with



(a) As-Received Plate of ZnS Showing Orientation of Ring Specimen with Respect to Direction of Vapor Deposition.



(b) Engineering Drawing giving Ring Specimen Specifications (All dimensions in mm)

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FIGURE 1 ZINC SULFIDE RING SPECIMEN

the inside surface of the specimen ring. The tensile strength σ_f is taken as the maximum hoop stress when the specimen fractures and is calculated from the hydrostatic pressure by the expression

$$\sigma_f = \frac{R_o^2 + R_I^2}{R_o^2 - R_I^2} P_c \tag{1}$$

where R_o and R_I are the outside and inside ring radii and P_c is the internal pressure at the time of fracture.

The expanded ring test technique is particularly suited for testing brittle materials, because it eliminates nonaxial stresses resulting from misalignment and localized stress concentrations caused by gripping or supporting the test specimen. The ability of this method to generate well-defined tensile stresses in the absence of any parasitic stresses is indicated by the dramatic increase in precision in tensile strength measurements in ceramic materials. Standard deviations of less than 3% compared with 10% to 50% for other test methods are typical for alumina and similar materials.

For fracture toughness determination, the ring specimens are provided with a starter notch on a plane parallel to the ring axis. Loading to failure is accomplished in the same manner as in tensile strength tests by increasing the pressure in the central annulus. Fracture toughness values are calculated from the recorded pressure and measured notch depth using available two-dimensional analytical solutions for cracks in internally pressurized cylinders.[3,4] For notched rings, these solutions have the form

$$K_{Ic} = A\sigma_c\sqrt{\pi a} \tag{2}$$

where K_{Ic} is the plane strain fracture toughness, σ_c is the critical hoop stress at notch instability,* a is the notch length, and A is a geometrical constant. The values of A for notches in rings of several geometries are given in Figure 2.

Results

Six expanded ring tests were performed at stress rates of 25 MPa/min. (or strain rates of 3.3×10^{-2} min^{-1}). Table 1 summarizes the data and results. Specimens 6 to 9 yielded consistent toughness values and indicated that the fracture toughness of CVD ZnS is 0.75 ± 0.01 MPa $m^{\frac{1}{2}}$. Two earlier tests performed on specimens having misaligned notches gave anomalously low values Table 2 compares the fracture toughness of several other brittle materials with the fracture toughness of ZnS. Zinc sulfide is similar in toughness to borosilicate glass and quartz, but significantly less tough than silicon nitride and alumina.

The crack emanated from a corner of the notch in all but one case (Experiment 9), and crack propagation was by a mixed transgranular/intergranular mode. The rings broke into three to five pieces. In addition to the crack at the notch, a crack always occurred diametrically opposite from the notch. The next most common fracture location was at 120^o and 240^o from the notch site.

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*For a notch extending inward from the outside ring surface

$$\sigma_c = \frac{2R_I^2}{R_o^2 - R_I^2} P_c$$

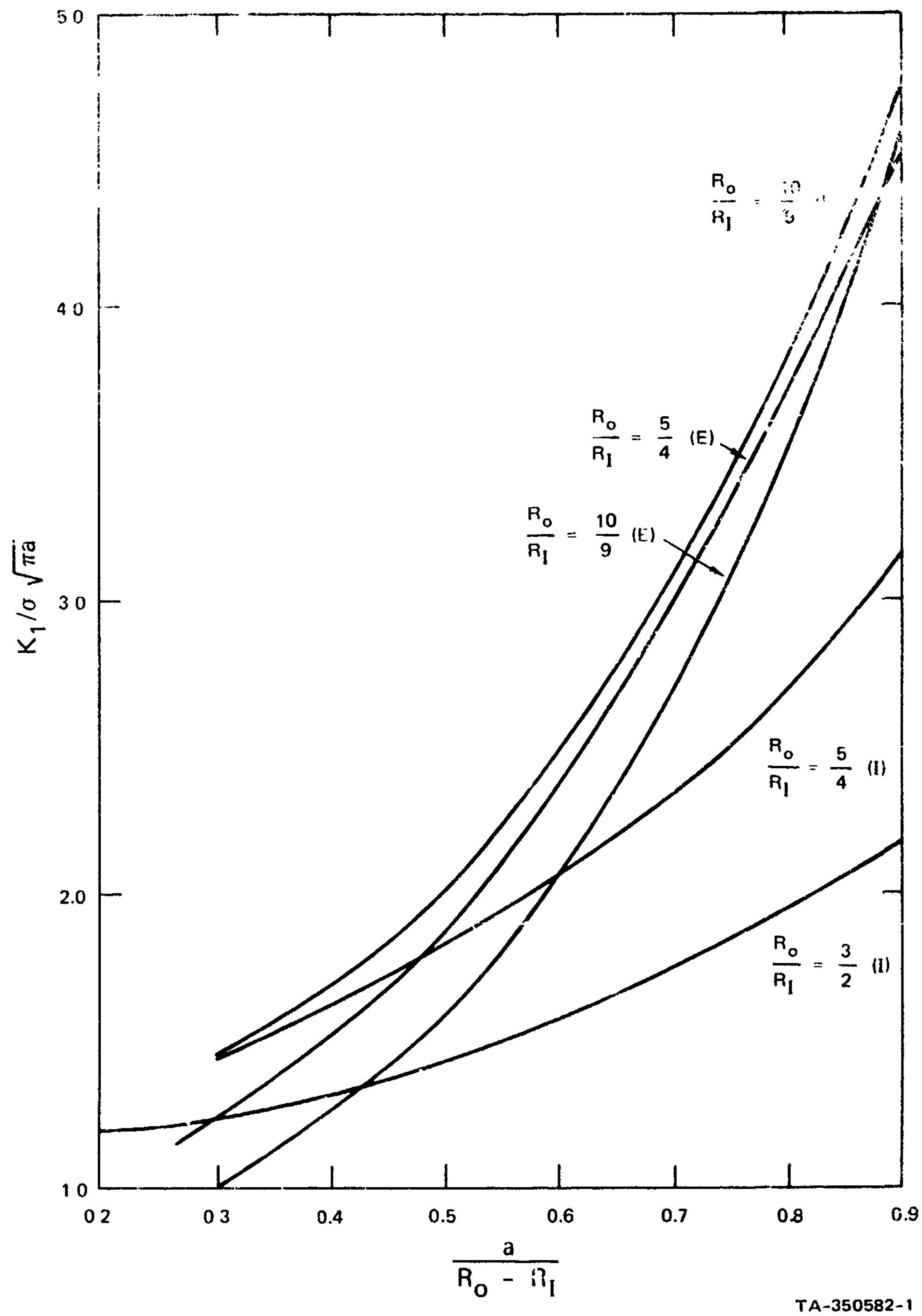

FIGURE 2 K-CALIBRATION CURVES FOR INTERNALLY (I) AND EXTERNALLY (E) NOTCHED RINGS OF VARIOUS RADIUS RATIOS, (R_o/R_I).

TABLE 1

EXPERIMENTAL DATA AND RESULTS FROM SIX EXPANDED RING TESTS ON CVD ZnS

Specimen No.	External Diam. D_o m x 10^{-3}	Internal Diam. D_I m x 10^{-3}	Wall Thickness $R_o - R_I$ m x 10^{-3}	Notch Size, a m x 10^{-3}	Burst Pressure P_c MPa	External Surface Stress σ_c MPa	Normalized Crack Length $\frac{a}{R_o - R_I}$	Calibration Factor (from Fig. 2)	Fracture Toughness K_{Ic} MPa $m^{1/2}$	Comments
1	30.50	25.36	2.57	1.22	1.15	5.15	0.475	1.68	0.54	misaligned starter notch
5	30.50	25.36	2.56	1.19	1.22	5.67	0.469	1.66	0.56	misaligned starter notch
6	30.52	25.36	2.57	1.22	1.67	7.45	0.475	1.68	0.77	
7	30.50	25.36	2.56	1.27	1.50	6.72	0.500	1.77	0.73	
8	30.50	25.35	2.51	1.19	1.63	7.28	0.676	1.67	0.74	
9	30.50	25.36	2.54	1.24	1.56	6.99	0.188	1.72	0.75	failed at midnotch

Wall thickness and crack size measured across the fracture face to 0.025 mm.

TABLE 2

FRACTURE TOUGHNESSES OF SEVERAL BRITTLE MATERIALS

Material	Fracture Toughness ($MPa\ m^{\frac{1}{2}}$)	Reference
CVD Zns	0.75 ± 0.01	present
Hot press Si_3N_4	4.5 - 5.0	6
Reaction bonded Si_3N_4	2.3	
Borosilicate glass	0.76	7
Quartz	0.74	
Lucalox Al_2O_3	0;74	8
$BaTiO_3$	1.05	

Discussion

Published flexural strength values for CVD ZnS as measured in 4-point bend tests are nominally 16,000 ksi, or 110 MNm^{-2}.[5] Using the fracture toughness result determined in this work and the expression for a semi-circular edge crack under uniaxial tension,[9] namely,

$$a_c = \frac{\pi}{2}\left(\frac{K_{Ic}}{\sigma_f}\right)^2$$

we compute the size of the largest flaw in the material a_c to be 0.73 mm. This is approximately 100 times the cross section diameter of the columnar grains.

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